EXHIBIT A

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Nanocrystalline Silicon Ink

Fabio Zurcher, Brent Ridley, Joerg Rockenberger

[redacted]

Project Weekly Report

Big Picture Objective

- Formulate Ink of Hydrogen-Capped Silicon-Nanocrystals (nc-Si:H)
- [redacted]

Key Results

- [redacted] Amines, Esters, Amides, Polyethers + Anionic Surfactants Good; All Solvents Can Yield “Stable, Milky” Dispersions of nc-Si:H
- Tested 16 Surfactants in Xylene With High-Power Ultrasound: 3 Show Almost Clear, Yellow “Solutions” Of nc-Si:H After 0.5 Micron Filtration. [redacted]

NC-SI:H - DEVELOPMENT PLAN

STEP	PURPOSE	EXPERIMENTS	OS	Done	[redacted]
Solvent Screen	1. determine compatibility of nc-Si:H w/ various solvents	1. Ultrasonicate nc-Si:H powder in solvent; check oxidation w/ FTIR	[redacted]	[redacted]	[redacted]
Surfactant Screen	1. Determine miscibility of surfactants w/ solvents	1. 1% surfactant solution; optical inspection	[redacted]	[redacted]	[redacted]
Dispersion Screen	1. determine suitability of solvents + surfactants to disperse nc-Si:H	1. Ultrasonicate 1% surfactant solvent solution w/ 0.1% nc-Si:H; Optical inspection + filtration	[redacted]	[redacted]	[redacted]
Ink Formulation	1. Formulate 5 wt% Si-NC:H ink	1. Refine formulation recipe varying agitation parameters, surfactant conc. etc.	[redacted]		[redacted]
[redacted]	[redacted]	[redacted]	[redacted]		[redacted]

Project Is On Track:

- **Dispersion Screen Is Finished**
- [redacted]
- **Evaluated 650+ Formulations so far…**

[redacted]

Risks Map

Issue	Mitigation Plan	Risk
Agglomerate Size Bigger than 20 nm	• Higher Ultrasound Power (smaller tip) • Longer Ultrasonication Times • Increased Surfactant Concentration • Apply Ultrasonication + Surfactant During Etch • Separate Larger Agglomerates By Centrifugation + Filtration • Use Multidentate Surfactants • Let's do photovoltaics…	H
Very Low Mass Loading (< 0.1 %)	• Drop-Casting Instead Of Spin-Coating • Increased Surfactant Concentration • Higher Ultrasound Power (smaller tip) • Longer Ultrasonication Times • Apply Ultrasonication + Surfactant During Etch • Use Multidentate Surfactants	H
Colloidal Stability	• Increased Surfactant Concentration • Use Multidentate Surfactants	H
Impurity Levels	• Keep Surfactant Concentration As Low As Possible • Choose Small-Molecule Surfactants • Oxygen-Free Surfactants	H
Film Density	• Keep Surfactant Concentration As Low As Possible • Choose Small-Molecule Surfactants	H

3rd Screen: Dispersion Of nc-Si:H

Goals:

- Identify Surfactants + Solvents Which Can Disperse nc-Si:H During Ultrasonication

3. Screening Summary

Surfactants			Solvents									
Name	#	Class	Butyl ether	Xylene	Decaline	Diglyme	Terpineol	Ethyl ethoxy propionate	Anisole	Limonene	DhT acetate	1-Dodecene
[illegible]lamine	1	amine										
D[illegible]ylamine	2	amine										
T[illegible]ine	3	amine										
N,N-[illegible]thyldodecanamide	39	amide										
So[illegible]monooleate	4	sorbitol										
Li[illegible] acid ethyl ester	5	fatty ester										
[illegible]	[illegible]	PEO										
[illegible] X-100	[illegible]	PEO										
[illegible] X-114	8	PEO										
[illegible] XL-80N	9	PPO										
Igepal CO 630	21	PEO										
Igepa[illegible]520	41	PEO										
Igepa[illegible]	22	PEO										
P[illegible] (ethylene glycol) monooleate	17	PEO										
[illegible] 80	18	sorb PEO										
A[illegible]	[illegible]	siloxane										
A[illegible]	[illegible]	siloxane										
[illegible]136	10	?										
Merpol A	13	?										
MerpolOJ	14	?										
Rhodo[illegible]	23	?										
K-S[illegible]52/MS	27	sulfonate										
K[illegible]XD-A503	29	polymer										
D[illegible] KS-873N	31	anionic										
Di[illegible] 7004	32	?										

= "good" (milky)

= "ok" (turbid or flakey)

= bad (precipitated or reacted)

PEP = poly ethylene oxide
PEP = poly 1-methyl ethylene oxide (propyl oxide)
sorb = sorbitol derivate

Main Conclusions:

- Solvent: everything works $\Rightarrow$ concentrate on xylene
- Surfactant: amines, PEO, esters, amides + anionic work

nc-Si:H Ink Formulation

Goals:

- Identify Surfactant Suitable For Dispersion of <0.5 Micron nc-Si:H Agglomerates In Xylen

Surfactant	After centrifugation	After Filtration	FT-IR Oxidation?
Hexadecylamine		pale yellow	
Dimethyldecylamine		colorless	
Trioctylamine		pale yellow	
4-Dodecyldiethylenetriamine	yellow SN	yellow	
N,N-Diethyldodecanamide	pale yellow SN	pale yellow	
Sorbitan monooleate		n/a	
Linoleic acid ethyl ester	pale yellow SN	colorless	
C12E5		faint yellow	
Triton X-100		faint yellow	
Triton X-114		colorless	
Triton XL-80N		colorless	
Igepal CO 210		colorless	
Igepal CO 520		n/a	
Addid 130		pale yellow	
K-Sperse 152/MS	yellow/orange SN	yellow	
Disparlon KS-873N dispersing agent	yellow/orange SN	yellow	

colored, transparent
milky
colorless

Main Conclusions:

- 3 Formulations give yellow, mostly transparent solutions after 0.5 micron filtration
- 2 are anionic surfactants!!!! How does that work???
- Si-Mass loading estimated to be ~0.01% ⇨ Surfactant-Si mass ratio: 500

[redacted]

Summary

Results

- All Solvents Can Yield Milky Dispersions of nc-Si:H
- Amines, Polyethers, Esters, Anionic Surfactants “Work”
- [redacted]

[redacted]

Milestone Weekly Schedule – nc-Si

Crit Path	Milestone	Who	Start	OS	CS	Done ?	Comments
nc-Si	FTIR + TEM of Supernatants	BR/FZ	08/19	08/21			verfiy identity of supernatants
nc-Si	Sonnication of Surfactants + Solvents as Control	FZ	08/20	08/21			verfiy identity of supernatants
nc-Si	search for multidentate + polymeric amines	FZ/BR	08/18	08/25			
nc-Si	DOE on sonication: power, time, surfactant concentration	FZ/BR	08/19	08/25			
nc-Si	Screen surfactants in pyrdine	BR	08/20	08/23			

EXHIBIT B

Surface Derivatization of Silicon Nanocrystals

Fabio Zurcher, Brent Ridley

[redacted]

Pros & Cons of the AIBN Reaction

Pros	Cons
Short reaction time (30min)	Product shows reoxidation
Reliable reaction	Yield is not exceptionally high
No obvious source of metal or halogen contamination	AIBN byproducts are difficult to remove
Reliable and relatively simple isolation/purification step	Product is not extremely soluble in aromatics
Product is a well defined, dry powder	
Product is very soluble in hydrocarbons and ethers	

Derivatization Reaction Flow

Step	Description
Derivatization	nano-Si stock (etch product suspended in xylene) + dodecene + AIBN + solvent (xylene). [dodecene] = 1M; [Si] = 0.25M; [AIBN] = 0.1M T = 120°C t = 30min
Filtration	Reaction product is filtered hot through a 0.2µm PTFE filter
Precipitation	Product is precipitated with cold MeOH and centrifuged to remove the SN
Wash	The precipitated product is washed with AcN to remove residual AIBN byproducts and then centrifuged
Dry	The remaining solid is dried overnight under Ar or N_2 flow

[redacted]

EXHIBIT C

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Silicon Film Formation From Nanocrystals

Joerg Rockenberger, Fabio Zurcher, Brent Ridley

[redacted]

Surface-Modified Si Nanoparticles

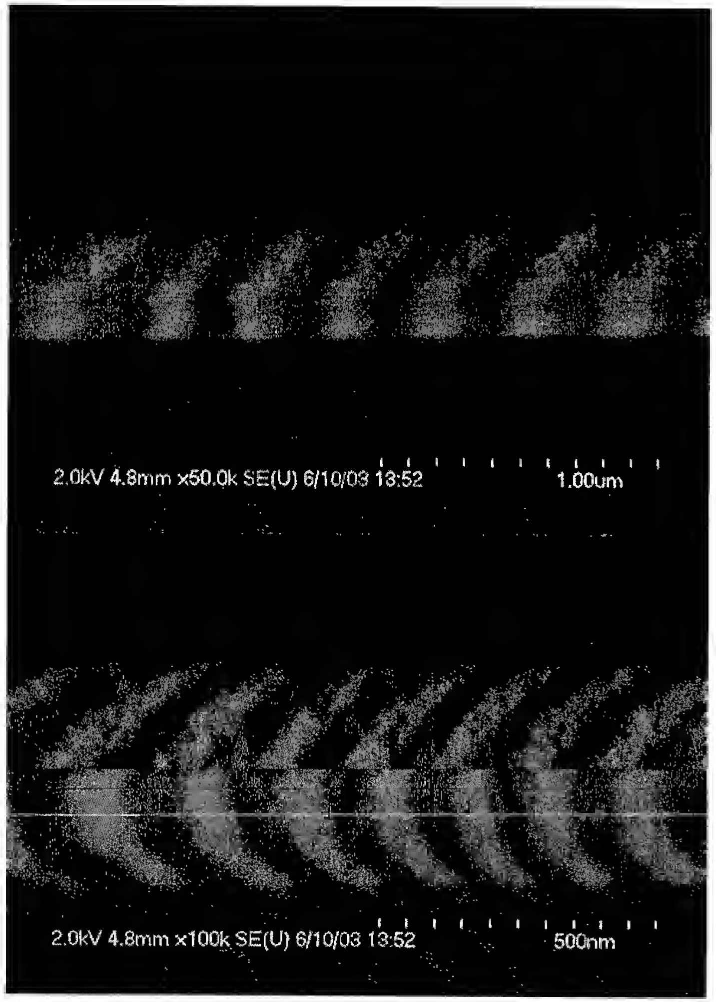
		AIBN	AlEtCl2
Synthesis:	Si-NC production [mg/batch]	30	90
	Synthesis + Isolation Time [h]	3	72
	Temperature [C]	120	40
Ink Formulation:	Oxygen – Level [%]	0.5	0.1 – 0.2
	TGA – Mass Loss [%]	20	TBD
	Solubility – Xylene [%]	TBD	5%
	Solubility – Butylether [%]	> 5%	< 1%
Film Characterization:	Oxygen – Level [%]	15 / 11	21
	Carbon - Level [%]	15 / 18	17
	Hydrogen – Level [%]	18	TBD
	SEM – Thickness [nm]	150 – 250	0 - 100
	SEM - Morphology	TBD	very rough – waffle
	XRD – Grain Size [nm]	TBD	TBD
	Tencor – Thickness [nm]	150 -250	85
	Tencor – Roughness [nm]	< 2	6

Biggest Difference: Related to Solubility + Resulting Film Morphology!

Surface-Modified Si Nanoparticles

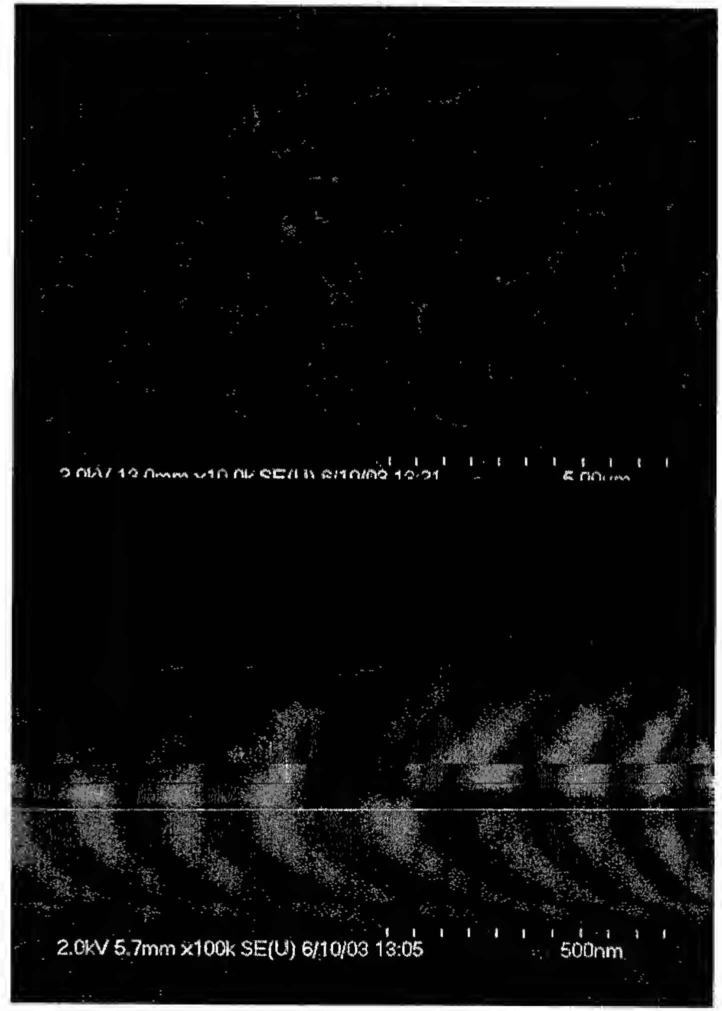
AIBN Reaction

5% in Butylether, 300 rpm
100 C softcure, 900 C hardcure



Lewis Acid Reaction

2% in Xylene, 300 rpm
100 C softcure, 500 C hardcure



[redacted]

EXHIBIT D

Polysilane ink formulation

[redacted]

Polysilane ink formulation

Goal

- **Find solvents that are compatible with cyclosilane.**
- **Reproducibly provide a polysilane ink formulation with a shelf life of > 1day.**

Module	Experiments	[redacted]
Ink formulation stability	Test cyclosilane compatibility in different solvents	
[redacted]	[redacted] 2. Thermally polymerize cyclosilane – FTIR – Solubility in cyclosilane, cyclooctane 3. UV polymerize cyclosilane at RT – FTIR – Solubility in cyclosilane, cyclooctane – Weight loss [redacted]	

Silane Compatibility With Solvents

Linear Alkanes	Cyclic alkanes	Alcohols/Ketones/Enes	Aromatics	Ethers	Fluoro
Decane	Cyclohexane	α–Terpineol	Benzene	Diethylether	Fluorinert FC 70
Tetradecane	Cycloheptane	DHT	Toluene	Butylether	Perfluoro(1-methyl)decaline
	Cyclooctane	1-Methoxy-2-propanol	O-xylene	Anisol	Fluorinert FC 70 + Perfluorooctansulfonylfluoride
	Cyclodecane (tbd)	α-Pinene	Mesitylene		
	Methyl-cyclohexane	2-Butanone	t-Butyl-toluene		
	t-Butyl-cylcohexane	2-Heptanone	Cyclohexyl-benzene		
	cis-decalin	Cyclopentanone	Tetraline		
	trans-decaline	Ethylpyruvate			
	Bis-cyclohexyl	Butylether			

stable > 2 days

Limited stability

white ppt.

white ppt., then dissolution

two phases

Polysilane ink formulation

Sources that may have an impact on ink stability	Parameters for experimental matrix
Light or temperature induced polymerization	Temperature: ~0C, 45C Light/dark: dark at 0C, clear/amber glass vials at 45C
Solvent purity (water and other impurities)	Distill and dry Cyclooctane, cis-decaline
OH (leaching from glass wall) induced polymerization	Teflon vials and silanized glass vials
Contamination from tips, cap lining	Use current pipettor tips, cap lining
Nature of silane mixture, SiH_3 groups as radical initiators)	Silane batch: fixed, [redacted]21-1A, needed: 30 x 30 uL = 900 uL; NMR control of cyclosilane/cyclohexane ink
Mass loading	Concentration: fixed, 20 vol%, Amount: fixed, 150 uL
Nature of solvent	Solvent: Cyclohexane, Cyclooctane, (Cycloheptane), Ether, toluene, (cyclodecane), decaline
[redacted]	[redacted]
[redacted]	[redacted]

EXHIBIT E

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Silane Ink Formulation

Solvent Selection and Controlled Polymerization

[redacted]

[redacted]

Solvent/Silane Compatibility

Solvent	bp (°C)	V (cP)	S (mN/m)	Cutoff (nm)	Solubility	FTIR	Thin Film
Cyclooctane	151	1.0			OK	No Si-O	**Alkanes/Aromatics**
cis-Decalin	193	3.0			OK	No Si-O (100 °C)	UV Film OK
Decalin (mixture)	191	2-3			OK		
o-Xylene	144	0.8	29.5		OK	No Si-O (100 °C)	
Tetralin	207	2.1			OK		
Methylnaphthalene	240	3.1			OK	No Si-O (150 °C)	
Tetradecane	252	2.1			Cloudy→OK	No Si-O (150 °C)	
D4 Cyclomethicone		2.4	17.4		OK	No Si-O (150 °C)	**Exotics**
D5 Cyclomethicone		3.8	17.4		OK	No Si-O (150 °C)	
Cineole*	176	2.3			OK→Solids	No Si-O (100 °C)	**Ethers/Alcohols**
EG-dibutylether	203	14			OK→Solids		
3-Octanol*	174	7			Cloudy	Oxidation	
2-Ethylhexanol	182	4-6			Cloudy	Oxidation	
Dihydroterpineol*	208	46			OK	Oxidation	
Dihydroterpineol (IFF)	208	46			OK→Solids		
Terpinen-4-ol*	212	12			OK	No Si-O (150 °C)	Film oxidation
Terpineol*	217	37	32		OK	No Si-O (150 °C)	Film oxidation
Pine Oil 60		5			OK	Oxidation	

***Similar results after drying solvent over molecular sieves**

Silane Polymerization

Sample	Loading (vol%)	Solvent	Exposure (min)	Observation (solution)	Observation (cast)	FTIR
UV			UV			
[redacted]4-60-0	none	c-C_8H_{16}	5min	Clear	N/A	N/A
[redacted]4-60-1	5%	c-C_8H_{16}	5min	Cloudy (from walls)	Good wetting/film	No change(s)
[redacted]4-60-2	5%	*o*-xylene	5min	Clear (even walls)	Good wetting/film	No change(s)
[redacted]4-60-3	5%	c-C_8H_{16}	20min	Clear	Good wetting/film	No change(s)
[redacted]4-62-1	25%	c-C_8H_{16}	20min	Clear	Good wetting/film	No change(s)
[redacted]4-62-2	25%	c-C_8H_{16}	60min	Milk	N/A	Broad, Baseline
[redacted]4-63-1	25%	*o*-xylene	40min	Cloudy	Good wetting/film	Broadening
[redacted]**4-68-1**	**100%**	**none**	**20min**	**Clear, viscous**		**Broadening**
[redacted]**4-68-2**	**100%**	**none**	**60min**	**Pale amber, viscous**		
Molecular Sieve			Sieves			
[redacted]4-58-1	5%	c-C_8H_{16}	none	Clear	Poor wetting/film	N/A
[redacted]4-60-2N	5%	*o*-xylene	none	Clear	Poor wetting/film	N/A
[redacted]4-58-1S	5%	c-C_8H_{16}	4days	Clear	Poor wetting/film	No change(s)

White films form routinely form on walls above liquid level (from vapor)

Viscous Silane(s) Solubility

Solvent	[redacted]26	[redacted]95-1	[redacted]2-10-1	[redacted]-68-1	[redacted]-68-2	[redacted]
Alkanes						
c-C_8H_{16}	OK*	OK*	OK*	OK	Turbid*	
cis-Decalin	Gloppy*	Turbid*	OK*	OK	Precipitate*	
Aromatics						
o-xylene		Turbid		Turbid		
Methylnaphthalene		Gloppy		Gloppy	Precipitate*	

*** 10% solutions – all others 20%**

Silane Ink Formulation [redacted]

Big Picture Objective:

- Formulate silane ink suitable for inkjet printing (viscosity, surface tension, etc.)
  1. Appropriate solvent selection
  2. Controlled silane polymerization
- [redacted]
- Investigate alternatives to inkjet technology (microspot…)

[redacted]

Key Results:

- The viscous tertiary alcohol terpinen-4-ol is retained in UV-spun silane films and leads to oxygen and carbon loaded silane films after curing at 400 °C
- After drying over sieves, alcohol and ether solvents still cause problems with solubility and/or oxidation – so far, no solvents containing oxygen have worked except for the cyclomethicones
- UV polymerization reactions initiated – control of viscosity is underway [redacted], but polymerization (and precipitation) occurs in both cyclooctane and xylene solvents

[redacted]

EXHIBIT F

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Solvent Compatibility Tables

Technical Report

[redacted]

Activated Alumina Purification for Solvent Screen - [redacted]

Purpose
- Purify inkjet solvent candidates and test solubility, stability, and printability

Method
- Single-pass column purification of solvent – 9g activated alumina (500 °C under vacuum 6h), collecting 4mL fractions in amber vials after discarding first mL
- 20% solution using [redacted] SOP polysilane

Solvents
- Alkanes: Tetradecane, dicyclohexyl, decane, pinane, *t*-butylcyclohexane, isopropylcyclohexane, trimethylcyclohexane
- Aromatic: Methylnaphthalene, tetralin, cyclohexylbenzene
- Ethers: Diethylene glycol diethyl ether, dibutyl ether
- Halogenated: Chlorooctane, cyclohexyl chloride
- Silane: Tetraethylsilane

Analysis Proposal
- Suggested testing, in order of execution: miscibility and solubility at 50% and 20%, stability at 20%, viscosity, contact angle, printability, NMR, GC-MS

Results
- All 15 solvents were poor solvents for polysilane!
- Most were turbid solutions or two phase mixtures
- Some solvents formed two clear phases – tetradecane, decane
- **What makes *cis*-decalin and cyclooctane (and cyclodecane) so good?**

Solvent/Silane Compatibility - 2004

	Solvent	bp (°C)	η (cP)	γ (mN/m)	Solubility (5-10 vol%)	FTIR	Thin Film
Alkanes	**Cyclooctane**	151	2.2		OK	No Si-O	TFTs
	Nonane	151	0.6	25	OK→Precip		
	Decane	174	0.8	23	OK→Precip		
	Decalin (mixture)	**191**	2.2	**32**	OK		
	***cis*-Decalin**	**193**	3.0	**32**	OK	No Si-O (100 °C)	TFTs
	Cyclodecane	**201**	**4.3**		OK	No Si-O (150 °C)	
	Dicyclohexyl	**207**	3.3		OK*		
	Dodecane	216	1.2	25	OK→Precip		
	Tetradecane	252	1.9		Cloudy→OK *	No Si-O (150 °C)	
Aromatics	*o*-Xylene	144	*0.8*	30	OK*	No Si-O (100 °C)	
	1,2 Dichlorobenzene	180	*1.3*	**37**	Cloudy→OK *	No Si-O (150 °C)	
	Tetralin	207	*2.1*	**33**	OK*	No Si-O (100 °C)	
	Methylnaphthalene	240	*3.1*	**40**	OK*	No Si-O (150 °C)	UV Film Patchy Streaks
Heteroatomic	2,6-Lutidine	144		**32**	*		
	Quinoline			43	OK	Si-O (150 °C)	
	Anisole	154		**35**	Cloudy (10%)	No Si-O (150 °C)	
	Phenylether	256		**39**	Cloudy (10%)	No Si-O (150 °C)	
	D4 Cyclomethicone		2.4	17	OK	No Si-O (150 °C)	
	D5 Cyclomethicone		3.8	17	OK	No Si-O (150 °C)	UV Film Patchy Streaks

*** Limited solubility above 5% or with polymeric silanes** ***Italicized viscosity values are from literature, not in-house measurements***

Solvent/Silane Compatibility (Problematic) - 2004

	Solvent	bp (°C)	η (cP)	γ (mN/m)	Solubility (5 vol%)	FTIR	Thin Film
Exotics	D4 Cyclomethicone		2.4	17.4	OK	No Si-O (150 °C)	
Exotics	D5 Cyclomethicone		3.8	17.4	OK	No Si-O (150 °C)	UV Film Patchy Streaks
Ethers/Alcohols	Cineole*	176	2.3		OK→Solids	No Si-O (100 °C)	
Ethers/Alcohols	EG-dibutylether	203	14		OK→Solids		
Ethers/Alcohols	3-Octanol*	174	7		Cloudy	Oxidation	
Ethers/Alcohols	2-Ethylhexanol	182	4-6		Cloudy	Oxidation	
Ethers/Alcohols	Dihydroterpineol*	208	46		OK	Oxidation	
Ethers/Alcohols	Dihydroterpineol (IFF)	208	46		OK→Solids		
Ethers/Alcohols	Terpinen-4-ol*	212	12		OK	No Si-O (150 °C)	Film oxidation
Ethers/Alcohols	Terpineol*	217	37	32	OK	No Si-O (150 °C)	Film oxidation
Ethers/Alcohols	Pine Oil 60		5		OK	Oxidation	

*Similar results after drying solvent over molecular sieves